

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of

Gerhard COUFAL

Serial No. 09/830,074

Filed April 23, 2001

METHOD FOR PRODUCING PURE
MELAMINE



Docket No. 2001-0462A

Group Art Unit 1624

Examiner V. Balasubramanian

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DECLARATION UNDER 37 CFR 1.132

I, Gerhard Coufal

declare that I am citizen of Austria, residing at Muenchgasse 21,
A-4060 Leonding, Austria;

That my education and employment history are as follows:

After finishing a polytechnical college in mechanical engineering, I worked for 1 year as a designing engineer at VOEST-Alpine in Linz, Austria.

Then I studied technical chemistry at the Technical University in Graz, where I received a M. Sc. (Dipl.Ing.) in 1981. In Sept. 1981 I joined Chemie Linz, a predecessor of Agrolinz Melamin, where I worked in the Research Department, being engaged in the development of a production process for 1,2,4 triazol.

Then I was production assistant in the acrylonitrile plant in Enns and from 1982 to 1983 in the urea/melamine plant in Linz. From 1990 to 1996 I was technical director of the chemical plants of Agrolinz in Castellanza, Italy, including the urea/melamine plant. Since 1996 I am again in the urea/melamine plant in Linz, where my main responsibility is the development of the new melamine technology of Agrolinz Melamin.

I am inventor/coinventor of numerous patents/patent applications in the field of synthesis, purification and application of melamin , for example of US 5,721,363, US 5,087,384, EP 799213, US 6,380,385, US 6,355,797, WO 00/32566, WO 00/29393, WO 00/39107,

WO 00/55142, WO 01/07420, WO 01/07421, WO 96/20182, WO 96/20183, WO 96/23788, WO 97/20826.

That I have conducted the following experiments to demonstrate that by cooling crude liquid melamine to a temperature just above its melting point followed by quenching with aqueous ammonia, purity and yield of the obtained melamine are higher than by quenching crude liquid melamine of higher temperature with aqueous ammonia.

Example according to the invention:

20 g of crude melamine having a melam content of 2 % by weight and a melem content of 1 % by weight were introduced into an autoclave having a volume of 100 ml, and the autoclave was brought to a temperature of 370 °C with an NH₃ feed at an NH₃ pressure of 280 bar and was kept at this temperature and pressure for 2,5 h. Cooling was then effected to 320 °C in the course of 1 h with an NH₃ feed, the pressure of 280 bar being maintained. The temperature of 320 °C was maintained for 30 min and then the melamine melt was sprayed into a second autoclave (1000 ml volume). The second autoclave contained 100 ml of a 10 % aqueous ammonia solution, the temperature being 160 °C, the pressure 12 bar. After spraying the autoclave was kept at these temperature and pressure conditions for 10 minutes.

After cooling of the autoclave, filtration and drying, 19,6 g melamine having a purity of 99,4 % was obtained.

Comparative Example:

20 g of crude melamine having a melam content of 2 % by weight and a melem content of 1 % by weight were introduced into an autoclave having a volume of 100 ml, and the autoclave was brought to a temperature of 370 °C with an NH₃ feed at an NH₃ pressure of 280 bar and was kept at this temperature and this pressure for 2,5 h. Then the melamine melt was sprayed into a second autoclave (1000 ml volume) which contained 100 ml of a 10 % aqueous ammonia solution, the temperature being 160 °C, the pressure 12 bar and kept at these temperature and pressure conditions for 10 minutes.

After cooling the autoclave, filtration and drying, 19,3 g melamine having a purity of 98,0 % were obtained.

The undersigned declarant declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001, of Title 18, of the United States Code and that such wilful false statements may jeopardize the validity of the application or any patent issuing therein.

Signed this 6th day of June, 2002



Gerhard Coufal